THE MANUFACTURE OF FLAVORING EXTRACTS.

By E. M. CHACE,

Assistant Chief, Division of Foods, Bureau of Chemistry.

NATURE OF FLAVORING EXTRACTS IN GENERAL.

The flavoring extract, as we know it, is a product peculiar to America. While all civilized countries are familiar with the flavors employed in the manufacture of flavoring extracts, few, if any, use them as they are used in this country. A flavoring extract, to quote from the standards established by the Secretary of Agriculture in 1906, "is a solution in ethyl alcohol of proper strength of the sapid and odorous principles derived from an aromatic plant, or parts of the plant, with or without its coloring matter, and conforms in name to the plant used in its preparation." This definition at once excludes all preparations which are not solutions in alcohol and eliminates the various forms of prepared flavored sugars used abroad.

Extracts were at first considered almost exclusively as pharmaceutical products, being used partly in medicines and partly in foods by housewives and confectioners. For many years formulas for the preparation of such extracts appeared in the Pharmacopæia, but these have now been omitted, as the demands of the food and confection trade far exceed all others.

The two principal flavors are vanilla and lemon, it being estimated that more than 95 per cent of the flavoring extracts manufactured are of these two varieties. With few exceptions the other flavoring extracts are artificial, it being impossible to manufacture an acceptable extract from the fruit itself. Orange, peppermint, and wintergreen extracts are among the exceptions to this rule, while the strawberry, pineapple, peach, and some others are always artificial.

ORIGIN AND CULTIVATION OF THE VANILLA BEAN.

There is at least three times as much vanilla consumed as of all other flavors together, and in all probability the consumer knows less of its origin than of any other material from which extracts are made, few being familiar even with the matured vanilla bean.

The plant from which this fruit is gathered is a native of the southeastern portion of Mexico, where it was used by the natives for the purpose of flavoring cocoa when that country was conquered by the Spanish under Cortez. It was first described by a Franciscan friar, Bernhardino de Sahagan, in 1575, and for years was supposed to be of great medicinal value. For this reason many attempts were made to cultivate it in Europe, but with absolutely no success. Its cultivation in tropical countries, however, has since attained great magnitude, but in all cases the product has fallen short of the superior flavor of the native Mexican bean. It is now grown commercially in the East Indies (especially Java), Réunion, Mauritius, and the Tahiti islands. The Mexican product, however, still retains its superiority and brings a considerably better price than any of its descendants, in some cases the transplanted bean being decidedly inferior, as in the case of those from Tahiti.

The plant itself is a vine belonging to the family of orchids, the Vanilla planifolia Andrews being the species usually cultivated. It thrives only in the Tropics, the mean annual temperature of the countries of its production averaging over 80° F., with an average rainfall of approximately 38 inches. Some credence seems to be given to the theory that the vine requires the dense shade of a tropical forest, owing probably to the fact that its support when growing wild was the forest tree. Trees are generally used as supports when the plant is cultivated, more, however, because they are readily available than for any other reason. The plants bear when three years old and continue bearing for thirty years or more. Little difficulty seems to be encountered in the cultivation of the vanilla bean, although the vine is subject to the usual attacks of insects and disease.

ARTIFICIAL POLLENIZATION OF THE VANILLA BLOSSOM.

An interesting incident in the growth of the fruit is the artificial pollenization of the flower. The original cultivators depended solely upon insects to carry the pollen from blossom to blossom. The results were naturally unreliable, some vines being made to bear too many pods, others too few, resulting in a loss in both quality and quantity of the fruit produced. To overcome this difficulty the modern planter pollenizes by hand. The process, an exceedingly simple one, is carried out by removing the pollen from the male flowers by means of a small wooden splinter about the size of a toothpick and placing small portions of it in the female flowers. (A similar method is used by producers of hothouse fruit in this country.) As the flowers open during the night and close before midday, this work is done in the early morning and is most successful if completed on the first day on which the flower blossoms. If not successfully pollinated, the flower soon withers and falls; thus the number of pools which each vine is maturing can be seen at a glance and can be controlled according to its age and strength.

HARVESTING AND CURING THE VANILLA BEAN.

The pods mature in from six to seven months after the flowering period, becoming slightly hard and turning yellow at the lower end, whence thin yellow stripes run upward. This is a critical point in the production of the high-grade bean, great experience being necessary in order to detect the proper state of maturity for gathering. If the pod is picked when too green, the flavor when cured is inferior, and, furthermore, the bean is susceptible to mold. When it remains too long upon the vine it splits while curing and sells at a lower price. At the gathering time the bean has neither an agreeable odor nor flavor, both qualities being developed by the curing process. When allowed to become fully dry upon the vine, the bean does develop an odor and a flavor, but both are so inferior to those obtained when it is properly cured that the product thus obtained is almost worthless.

A different curing process seems to be in use in each locality; indeed, in most cases each planter has his own particular method. There is some disagreement among experts as to the exact nature of the change which vanilla undergoes in the curing process. By some it is contended that the change is merely one brought about by drying, while others assert that the bean undergoes a fermentation not unlike that to which cocoa is subjected. The simplest method in use is that common in Mexico, where the pods are allowed to lie in the sun for several hours until thoroughly heated, when they are wrapped in blankets and left until the following day. The heating, followed by storage, is repeated on several successive days until the greater part of the moisture has been evaporated. This procedure is, of course, often varied, it being a common practice to coat the beans with secret preparations, composed principally of animal or vegetable oil, to promote the sweating which takes place while wrapped in The excess of moisture is sometimes removed by manipulating the beans with the hand, and many other devices are used to develop the desired flavor. In the French colony of Réunion the beans are subjected to a scalding bath immediately after picking; thus wilted, it is claimed that the subsequent fermentation, desiccation, and manipulation are greatly aided.

In all localities artificial drying has now largely replaced exposure to the sun, thus shortening the time consumed in curing and giving a more even product.

GRADING AND STORAGE OF VANILLA BEANS.

When the beans are finally ready for shipment they are sorted according to length, those which have split or become otherwise defective being separated. The former are sold as "splits;" the latter, after having the defective portion removed, are known as "cuts,"

both being lower priced than the whole bean. The beans when properly prepared and stored may be preserved for several years. After a short period of storage the East Indian varieties become covered with a white coating, or what is generally known as a frosting of vanillin crystals. A like crystallization takes place with the Mexican varieties, but to a much less extent, for, while of superior flavor, the Mexican bean contains less vanillin. Although it is generally conceded that the principal flavoring agent of all varieties is vanillin, it is certainly true that other compounds give the fine bouquet to the Mexican bean which enables it to command the highest market price. The beans, which are now dark brown, are purchased and stored in large quantities by the dealers in this country. It is necessary to go over the stored material every few weeks and remove the bundles which show signs of mold or other infection. (See Pl. XXVI, figs. 1 and 2.)

THE MANUFACTURE OF HIGH-GRADE AND LOW-GRADE VANILLA EXTRACTS.

The task of converting the bean into extract of the first quality is exceedingly simple. An ounce of the beans, finely cut by machines, is allowed to soak in 10 ounces of a mixture consisting of equal parts of grain alcohol and water. In the majority of cases the extract is poured off from the exhausted beans in a few days, bottled, and shipped to the retail dealer. A very few manufacturers allow the mixture to remain for months in casks which have been used for this purpose many years. The theory that this treatment produces a superior bouquet is often denied, but the producers of the highest grade of extract still continue its use, despite the fact that the loss through evaporation and the delay in returns from the capital invested is considerable.

Cheaper extracts are made from low-grade beans, using less alcohol, the flavor and body of the product obtained being inferior. Small quantities of essential oils, and even musk, are used to supply flavor, and glycerin and sugar are added to give body. The latter substances are allowed by the standards, sugar being required by the United States Pharmacopæia formula.

In testing vanilla extracts the chemist bases his judgment of the purity of the sample largely upon the amount of vanilla resins which are present. Many of the cheaper grades are made in alcohol so dilute that the resins of the bean are not dissolved, and in order to produce the required amount various expedients are used. One of the most common is to heat the bean under pressure with glycerin; another is to treat the resins with alkali, rendering them soluble. Not a few manufacturers make use of the various forms of soluble oleo-resins of vanilla which are manufactured by large drug houses.

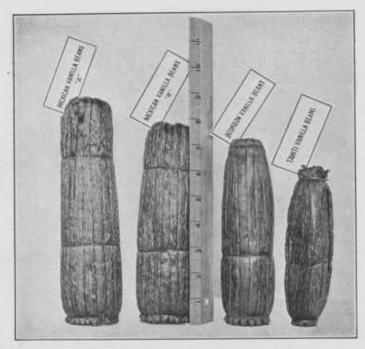


Fig. 1.—MEXICAN, BOURBON, AND TAHITI VANILLA BEANS.



FIG. 2.—BOURBON SPLITS AND BEANS, SHOWING FROSTING.



FIG. 1.—CUTTING THE LEMONS, MASCALI, SICILY.



Fig. 2.—Removing the Pulp of Lemons, Mascali, Sicily.



Fig. 1.—Expressing Lemon Oil, Two-piece Method, Mascali, Sicily.



Fig. 2.—Expressing Lemon Oil, Two-piece Method, Messina Sicily.



Fig. 1.—Interior of Lemon-oil Factory, Near Messina, Sicily.



FIG. 2.—LEMON-OIL MACHINE, REGGIO, CALABRIA.

The cheapest form of extract contains no vanilla, being made from the artificial vanillin, extract of tonka, or artificial coumarin. All of these products have strong odors and flavors, but none of the bouquet of the genuine bean. Such extracts usually contain prune juice, caramel coloring, sugar, glycerin, and other products to modify the flavor. The Federal food regulations prohibit the entry of such products into interstate commerce unless labeled "Artificial," "Imitation," or "Substitute," and almost all State laws require the same labeling.

SOURCES OF LEMON OIL.

Lemon extract ranks second only to the vanilla in point of the quantity consumed. It is made by dissolving the oil of lemon (5 parts) in strong alcohol (95 parts). Oil of lemon is the essential oil secreted by cells lying near the outer surface of the lemon rind. The similar oil found in orange peel is better known and to it is due the burning sensation felt when the skin is placed in the mouth. The world's supply of lemon oil comes from the island of Sicily, situated off the southwestern point of Italy, in the Mediterranean Sea. This island is the greatest lemon-producing region of the world, and large quantities of the fruit are shipped from it to all parts of Europe and to the eastern section of North America. The oil is produced as a by-product of this industry from the cull lemons. When the fruit is gathered it is carefully sorted and the lemons which for any reason will not stand shipment are sold to the lemon-oil factories.

THREE PROCESSES FOR EXTRACTING LEMON OIL.

There are three different processes in use for obtaining the lemon oil, two of which are known as sponge methods and the third as the machine method. The two-piece sponge method is the one generally used, in which the fruit is cut in half before separating it from the pulp. In the other the rind pared from the fruit is in three pieces. Less than 5 per cent of the lemon oil produced is made by machine, the use of which is confined to the coast of Calabria, on the Italian mainland.

THE TWO-PIECE SPONGE METHOD.

In the first-mentioned method the lemons are cut in halves by children or women, the cheapest form of labor. An ordinary paring knife is used, and the fruit is divided from end to end or crosswise, the former method being employed usually only when it is intended to make some use of the skin after extracting the oil. The work is carried on with great rapidity, the knife being started through the rind and the lemon cut in two and thrown into storage tubs by one motion of the arm. The tubs containing the cut fruit are dumped

into shallow troughs, where the pulp is separated from the skin. Older girls and women are employed for this work almost exclusively. A spoon-shaped instrument is inserted between the pulp and rind, and with one twisting motion forced toward the end of the fruit, when, with a quick jerk, the pulp is pulled from the rind and deposited in the trough. The rinds are thrown into baskets, and before being carried to the spongers are thoroughly soaked in water, usually by immersing the entire basket in a large tub or reservoir, and shaking off the excess. They are then allowed to stand four or five hours, or even over night. (See Pl. XXVII, figs. 1 and 2.)

The work of expressing the oil is done entirely by men, women being rarely employed, as the work is quite laborious. The workers sit upon low stools, the skins being dumped upon the floor in front of them and a basket for the exhausted skins set a little to one side. A small earthenware bowl, 8 to 10 inches high and of about the same diameter, is placed on the floor between the workman's knees. This bowl has at one side a lip, directly beneath which is a small concave depression, which serves to hold back the residue when the oil is poured from it. Across the top is placed a round stick of wood about an inch in diameter, so notched as to fit the widest part of the bowl. Across this stick is hung a flat sponge surmounted by another thicker one and finally a third, which is cup-shaped, into which the lemon skin is inserted with the right hand, the left being used to press upon the sponge, the weight of the whole body being thrown into the motion. The lemon rind is then turned partly over and the pressure renewed. This is repeated three or four times, after which the skin is thrown into the waste basket. Each half rind is handled separately, receiving three or four pressings. (Pl. XXVIII.) 1,600 to 2,200 of these halves produce only 1 pound of oil, the quantity depending upon the size, ripeness, and freshness of the fruit. It is said that green fruit produces rather more oil than ripe, and that the lemons should be worked up as soon as possible after picking. A good workman can produce between 2 and 3 pounds of oil per day, for which he receives from 40 to 60 cents.

By the two-piece method only a small quantity of water is expressed with the oil and the process of separation is very simple, the bowl being tilted forward until the oil can be blown from the surface over its edge into another receptacle. The water and residue remaining are separated from the traces of oil by the same means and finally filtered through felt bags. The residues left in these bags are collected for several days, when the bags are placed under a hand press and freed from the last traces of oil. The oil resulting from the filtration of the residues (called "fece") is of very low quality, with a decidedly disagreeable odor. It is not sold separately, but mixed with large consignments of the pressed oil.

THE THREE-PIECE SPONGE METHOD.

The three-piece method differs from the one just described mainly in the preparation of the skins before pressing. The rind is pared off in three slices, leaving the greater part of the pulp with some little rind at the ends. The paring, as a rule, is done by boys or men and the skins are washed or soaked, as in the two-piece method. method of sponging differs only slightly; the pieces being smaller are not inserted in a cup-shaped sponge, but are pressed flat against a large sponge placed over two others, as in the first method. earthenware bowl is always used and sometimes the oil is pressed directly into it: in other cases it is supplemented by setting a white glazed bowl on top of it. The sponge stick is fitted to this and the oil received directly, the larger bowl being then used only for the separation of oil and residual juice. Much more pulp is left adhering to the skins by this method of procedure and therefore much more juice is expressed with the oil than by the other method. is made, however, that oil made in this way filters more rapidly and remains clear longer. The explanation given is that, more of the oil-soluble materials being coagulated by the citric acid, they are more easily removed and do not precipitate later.

MACHINE METHOD.

The use of machines in producing lemon oil is confined to the Province of Calabria, the oil thus produced forming but a very small part of the total product. It has more color than the sponge oil and is used to deepen the color of the latter when produced late in the season. The machine is extremely crude. The lemons, about eight in number, which must be of a uniform size, are placed in the receptacle between the grinding disks, the lower of which is stationary, while the upper one is turned by an arrangement of wooden cogs against the side flywheel. The pressure exerted by the weight of the upper disk is partly compensated for by the arm at the rear, which is also used to raise this part of the machine, so that the fruit may be placed in position and removed. A small bell rings after a given number of revolutions, usually about one-half minute being required to remove the oil-bearing part of the lemon skin. The fruit is then removed and carefully wiped with a sponge, the greater part of the oil and gratings having been collected in a receptacle placed under the lower The mixture is filtered through cloth filtering bags, the water and oil being separated by blowing the latter from the top. filter bags containing the final residue are pressed under hand presses similar to those used in Sicily. (Pl. XXIX.) The oil produced is of a deep rich yellow color and is used solely for the purpose of bringing up the color of pale oils. It is finally filtered through filter paper, stored, and shipped in copper containers.

MANUFACTURE OF THE EXTRACT FROM LEMON OIL.

The process of making the extract from the oil in the case of the ordinary extract is extremely simple. The oil is dissolved in strong alcohol in the proportion of 5 parts oil to 95 parts alcohol; it is then filtered and bottled. Sometimes a small amount of coloring is added, as this solution has but a faint yellow tint. The formula of the Pharmacopæia prescribes lemon peel for coloring, but unfortunately the color thus obtained fades in the course of a few weeks, so that the trade has turned to other sources, using chiefly turmeric and anilin dyes. The chief cost in the production of such an extract is the alcohol, which must be relatively strong (not less than 85 per cent pure) in order to retain the 5 per cent of lemon oil in solution. With lemon oil at \$1 per pound and alcohol at \$2 per gallon, the latter represents over 90 per cent of the cost of material.

METHODS OF PRODUCING LOW-GRADE LEMON EXTRACTS.

The cost can be practically halved by the production of a terpeneless extract, which can be made in three ways: (1) By the solution in dilute alcohol of the so-called terpeneless oil of lemon; (2) by solution in strong alcohol of oil of lemon and then diluting and removing the oil which separates out; and (3) by washing lemon oil with dilute alcohol. The first method is seldom used; the second and third have been quite common, each having some advantage over the other. By each of the latter treatments the principal flavoring agent of the oil, the citral, is removed and there remain undissolved the terpenes, which constitute about 90 per cent of the oil. The terpenes as thus obtained still retain some citral, and have therefore some flavoring value. It is often claimed that they are made up into extracts and sold to bakers. Many of the cheaper products on the market are merely weak alcoholic washes made by repeatedly shaking the oil with dilute alcohol (about 20 or 30 per cent pure). They have something of the odor of a good extract, but are worthless when used for baking purposes. At times these extracts are flavored with citronella, and strengthened with lemon-grass citral containing glycerin, sugar, and other substances to give body and flavor.

ORANGE EXTRACTS.

True orange extracts are made by dissolving oil of orange in strong alcohol. This oil is manufactured by a process identical with that used for lemons, and practically the whole output, as in the case of lemon oil, comes from Sicily. The same problems of manufacture are encountered as with lemon extracts, but the production of cheaper grades is not so extensive, the demand for orange extract not being sufficiently large.

PEPPERMINT AND WINTERGREEN EXTRACTS.

Almost the sole use of peppermint and wintergreen flavors is in confectionery, and but few extracts appear on the general market. Both of these flavors are products of American soil. Peppermint is grown largely in southern Michigan and northern Indiana and in Wayne County, N. Y. Wintergreen is produced chiefly around White Haven, Pa., although there are other districts which distill quite large quantities. The methods of production for these oils are very similar. In the case of peppermint, which is a cultivated crop, the plant is moved and placed in large vats. These vats are provided with false perforated bottoms and are capable of being tightly closed at the top. After the mint plants are placed in the vat they are thoroughly trodden down, the top is fitted on, and steam is turned into the false bottom. The steam ascends through the mint in the vat and is carried off through a pipe at the side. This pipe is run through a trough of cold water or some other form of condensing apparatus in which the steam and peppermint oil are condensed. The resulting liquid consists of two layers, the lower or water layer being automatically drawn from the bottom of the receptacle and the upper layer of oil finally freed from the last traces of water by filtration through cotton.

In the distillation of wintergreen the plant is placed in a copper kettle containing water, fitted with a top connecting with a worm still. The water is boiled off and the vapors condensed. In this case the distillate also forms in two layers, the lower of which is wintergreen oil. It is separated by removing the upper layer of water and filtering through cotton. The extracts of both peppermint and wintergreen oils are made by dissolving them in alcohol. As placed upon the market they are usually artificially colored, as the pure extracts are almost colorless. Oil of birch and synthetic methyl salicylate are used largely to replace wintergreen oil in such products.

ALMOND EXTRACT.

This flavor is prepared by making a solution of oil of bitter almonds in strong alcohol, and in order to comply with the official standards must contain at least 1 per cent of the flavoring material. Almond oil is derived principally from the seeds of the apricot, although considerable amounts are obtained from almonds and peach kernels. The oils obtained from these different sources are very similar and are universally known as oil of bitter almonds. In its preparation, the kernels are ground and subjected to high hydraulic pressure in order to free them from the fatty oil which they contain. The residues are then reground, fermented, and finally distilled with steam. The resulting product contains a highly poisonous substance,

hydrocyanic acid, which must be removed before it becomes available for the preparation of extracts. This is accomplished by treatment with lime and copperas, which reagents remove the last traces of the impurity.

Artificial extracts are prepared from synthetic benzaldehyde, a coal-tar product. The flavor of both products is that often obtained

by the housewife by the use of bruised peach leaves.

IMITATION EXTRACTS.

As has been said, it is impossible to prepare several of the common flavors from the original fruit. When such is the case, resort is had to the synthetic product most nearly corresponding to the genuine flavor. This flavor in most cases is due to a class of chemical bodies known as esters or ethers which are produced in the growth of the plant. Commercially, the same bodies are manufactured from fusel oil and other higher alcohols. Each flavoring-extract manufacturer has his own secret formula for the preparation of each class of extracts; the predominating ester in each case is, however, usually the same, acetic and butyric ethers being most commonly employed. This class of extracts is usually colored with coal-tar products, and the Federal and most of the State laws require that they be labeled "Imitation," "Artificial," or "Substitute."